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Chapter 4

Adhesive Properties of Metals and Metal Alloys

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Abstract

The paper presents the effect of some surface treatment on the bonded joints strength of selected construction materials, adhesive properties of adherends after surface treatment and surface roughness. The aluminium alloys sheets, the titanium sheets and the stainless steel sheets were tested. In the experiments the following surface treatments were investigated: degreasing (chemical cleaning), mechanical treatment, mechanical treatment and degreasing, etching, anodising and chromate treatment. Adhesive joints were formed with a two component epoxy adhesive, Loctite 3430. Adhesive joint tensile-shear strength tests were performed in accordance with EN DIN 1465 standard on Zwick/Roell Z100 and Zwick/Roell Z150 testing machines. Adhesive properties were determined by surface free energy and surface free energy was determined by the Owens-Wendt method. The roughness of specimens was qualified by the method for measuring contact roughness, using an M2 profilometer manufactured by Mahr. The surface view was obtained by used NanoFocus uscan AF2. Results obtained from adhesive joint strength tests of materials evidence that surface treatment plays an important role in increasing strength of analysed joints. Tests indicate that in numerous instances this is mechanical treatment only or mechanical treatment followed by chemical cleaning which translate to the highest joint strength. The surface treatment method which introduces extensive changes in the analysed materials surface geometry is mechanical treatment. The results of surface roughness parameters measurement carried out on test samples subjected to anodising indicate that anodising has an impact on the height of surface irregularities. The application of various surface treatments in different structural materials allows modification of their adhesive properties, determined by the surface free energy. It was noted that different surface treatments contribute not only to the surface free energy changes but to the SFE components share in the total value. In the majority of variants of EN AW-2024PLT3 aluminium alloy sheet surface treatment the dispersive

component amounted to the 93-99% of the total surface free energy. The assumption then should be that in order for the determination of a particular surface for adhesive processes to be comprehensive it should account for the adherends surface geometry as well as its adhesion properties. The geometry of surface can influence the mechanical adhesion and the surface free energy is connected with both mechanical adhesion and the other constituent of adhesion – proper adhesion.

Keywords: adhesive properties, surface free energy, contact angle, metals, metals alloys, surface treatment

1. Introduction

Adhesive bonding provides an invaluable alternative to other modern methods of joining structural materials [1-3]. At present, bonding technology offers a range of applications for a number of various branches of industry, including in building, automotive, aircraft, machine-building, packaging manufacturing, and marine applications [4]. Bonded joints find applications in various structures and constitute an extensively used form of adhesive joint [5]. Adhesive bonding technology offers numerous advantages over other methods, and joining materials of dissimilar physical or chemical properties is a prominent mark of superiority. This feature frequently determines that adhesive bonding is the only applicable method, particularly in the case of adherends of different chemical composition and physical properties, which could pose a considerable problem if, e.g., a welded joint were to be applied.

The increasing popularity of adhesive bonding as a method for joining metals is a result of several factors [1-10], e.g., high joint strength and lack of stresses within the joint, along with low cost per unit resulting predominantly from the amount of adhesive used to form a single joint. Further advantages of adhesive joints are as follows: vibration damping, forming the joint without machine tools, expensive equipment or materials (nevertheless in certain cases the cost of technological instrumentation may prove to be high), lack of electrochemical phenomena usually accompanying other methods of joining metals, and the joining of dissimilar structural materials, frequently of substantial disproportions in geometric dimensions [2, 5]. Currently, adhesive joining is frequently applied in bonding polymer composite and metal substrates. Structural adhesive bonding is an indispensable method of joining thin-walled elements of sandwich construction, whose advantages are lightness and rigidity, which are essential properties in aircraft constructions [1, 3].

One requirement of structural bonded joints is proper strength [2, 3, 5]. This is important due to the fact that one of the basic requirements to be fulfilled by an adhesive joint is obtaining the desired static strength. The strength of adhesive joints is determined by several major factors: technological, structural, material and environmental [6, 7, 11-19].

Adhesive bonding technology comprises several consecutive technological operations: surface treatment, preparation and application of adhesive, joining substrates, cure conditioning,

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finishing and joint quality control [2, 3, 5]. Individual operations may consist of a number of stages of specific technological parameters; they might also require various items of equipment and instrumentation. Detailed conditions of adhesive bonding operation are selected based on, inter alia, the type of substrate, the geometry of elements and structures, joint formation conditions, production type, etc. [1-4, 7, 14, 18, 20, 21].

A summary of the previous studies of some issues in adhesive joints and bonding technology is presented in Table 1.

Issues in adhesive joints and bonding	
Influence of factors on adhesive joints' strength	Technological factors, e.g., surface treatment [1-4, 6, 9, 10-12, 17, 19]
	Structural factor [2, 3, 14, 18, 21]
Advantages of adhesive joints and bonding	Advantages of adhesive joints and comparison of bonding with other joining method [2, 3, 5, 20]
Adhesive properties	Surface free energy [12, 13, 19, 22]
	Wettability [19, 22]

Table 1. Summary of some issues in adhesive joints and bonding technology

The subject of the test and analysis were the issues of surface treatment, particularly the influence of surface treatment on surface free energy and strength of adhesive joints, and also the influence on the geometric structure of adherends.

2. Adherend surface treatment for adhesive bonding

2.1. The aim of surface treatment

Surface pretreatment is one of the first and most important technological stages in the adhesive bonding process. It is preceded by the analysis of properties, type and geometrical structure of a material surface for adhesive bonding, as the choice of an appropriate surface pretreatment method depends on these data [4-8]. In adhesive bonding, the surface of joined elements is defined as the part of the material where interactions with an adhesive occur [2]. This is connected both with the area and depth of interaction. In order to produce strong adhesive joints, surface pretreatments for adhesive bonding should ensure the following [3, 5, 13, 20]:

- removal of all contaminants that could significantly decrease adhesive joint strength (such as lubricants, dusts, loose corrosion layers, micro-organisms) from surfaces to be bonded,
- good surface wettability,
- repeatability of properties,

- correct surface development,
- good activation of surfaces of elements being bonded.

Surface treatment includes, inter alia, the following operations [5, 7, 23-26]:

- removal of surface contaminants, residues of technological processes (e.g., stamping, rolling, forging or machining) or of a protective layer providing, e.g., corrosion protection in storage and transport, and in the case of polymers, removal of additives migrating to the surface layer,
- changes in the geometric structure of the surface, through, e.g., increasing the surface roughness, which results in the development of a "true" wetting surface, the contact surface of, for instance, adhesive or paint,
- reduction of intermolecular forces' range, leading to the increase of the surface free energy, which is of significance to the wetting process.

Critchlow et al. [27] underline that a particular pretreatment for structural bonding will ideally produce a surface which is free from contamination, wettable by the adhesive, highly macro- or micro-rough, mechanically stable, and hydrolytically stable.

Spadaro, Dispenza and Sunseri [28] systematized surface treatment operations when presenting test results of the impact of surface treatment operations on adhesively bonded joints of aluminium alloys.

The selection of surface preparation method, including the choice of proper technological operations aimed at developing a desired structure and energetic properties, is dependent on multiple factors, predominantly the type of materials to be joined with an adhesive bond.

In the test of adhesive properties and adhesive strength the following materials were used:

- aluminium alloy sheets: EN AW-2024PLT3 and EN AW-7075PLT0,
- titanium sheets: CP1 (Grade 1) and CP2 (Grade 2),
- X5CrNi181 stainless steel.

The following surface treatments were tested:

- degreasing (chemical clearing) with degreasing agents
- mechanical treatment with abrasive tools ,
- anodizing,
- chromate treatment,
- etching,
- a combination of selected aforementioned methods.

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2.2. Characteristics of some surface treatment methods

2.2.1. Degreasing

Chemical cleaning of a surface, frequently referred to as degreasing, constitutes, in the majority of instances, the first surface treatment operation in the preparation of adherends for adhesive joining [5]. The surface of adherends is covered with a layer of grease, dust, various machining residues, or organic and non-organic substances. That is why chemical degreasing is applied to remove from the surfaces of adherends all contaminants which could decrease the strength of the adhesive bond in the adherends' contact area [20]. The selection of chemical cleaning method and degreasing agent is contingent on numerous factors, such as: the efficiency of chemical cleaning, the dimensions of adherends, the type of material, technological equipment available, etc. [2]. The degreasing process can be conducted with different degreasing agents and instrumentation [2, 3, 5].

The following cleaning methods are used in industrial manufacture [5]:

- solvent wipe,
- immersion in an ultrasonic solvent bath,
- vapour degreasing with solvent,
- washing with aqueous solutions,
- high-pressure water steam cleaning.

The most frequently applied degreasing agents include: acetone, petrol, benzene, ethanol, trichloroethane, tetrachloroethane, toluene, methyl ethyl ketone (MEK), to name but a few [2, 5]. A variety of cleansing agents such as organic solvents and water-miscible detergents are commonly available. The chemical cleaning procedure is carried out in immersion washers, treatment chambers or with a soaked cloth [5].

2.2.2. Mechanical treatment

Mechanical methods find applications in both the removal of contaminants and the changing of surface geometry [29]. The structure of the surface layer is a feature heavily dependent on the surface roughness. Although mechanical treatment acts towards changing the surface structure, and therefore towards its development, it fails to activate it for bonding.

Methods of mechanical treatment, employed as preparation of the adherend surface, include the following: abrasive tool treatment, abrasive blasting, grit blasting, peening, brushing, scraping and sanding [5, 11, 30, 31].

When employing these treatment methods, particular attention should be given to surface roughness geometry [32] so as not to generate excessive internal stresses, which could contribute to lowering the adhesive joint's strength. L.F.M. da Silva et al. [33] highlight that considerable surface roughness might result in increased stress concentration and consequently lower joint strength. Extreme surface roughness of adherends can lead to lowering of the

adhesively bonded joint's strength when the adhesive fails to penetrate and wet the surface irregularities. Analysis of surface topography with regard to the wetting angle proves that irregularities in the shape of elongated ridges, characterized by low apex angle value and high accumulation, are the most advantageous. It has been shown in the literature [21] that the highest joint strength properties are obtained when the surface roughness parameter of adherends (Maximum Profile Valley Depth) $R_m = 7 \div 25$ μm.

Some researchers argue that mechanical methods applied in surface treatment generate non-axial shear stresses, which lead to structural micro-cracks, e.g., micro-stresses, dislocations, sharp edges, crevices, etc. It was, furthermore, concluded that mechanical treatment can cause compressive stresses in the surface layer of adherends and, consequently, their plastic deformation. This, in turn, is a factor introducing stresses into the adhesive layer, which could reduce adhesive joint strength by 10-50 % [21].

2.2.3. Chemical treatment

Surface preparation with chemical treatment methods is employed in the case of adherends of a substantial amount of surface contamination. Chemical methods, based on liquid chemical compounds, enable surface and surface layer development. Their chemical constitution ensures high physicochemical activity of the surface with a bonding agent (e.g., adhesive), applied or medium substance [2, 5].

Aluminium alloy etching [21] showed that the constitution of etching bath has a great impact on the adhesive joint's strength; for instance, shear strength of aluminium alloys subjected to etching in 4 % NaOH is 40 % higher than in the case of 20 % HNO_3.

2.2.4. Electrochemical treatment

Anodizing is a widely used surface treatment operation applied in metals, consisting in electrolytic formation of oxide film. Anodizing finds applications predominantly in aluminium and its alloys; however, it may be used in certain types of steel, titanium and magnesium alloys. Eloxal process (electrolytic oxidation of aluminium) is a term frequently encountered in reference to aluminium anodizing [34].

Anodic oxidation of aluminium, known as anodizing, is a process during which on the surface of metal a thicker oxide layer is formed, providing superior corrosion protection to the natural passivation layer [35-37].

The anodizing process consists in the aluminium surface being transformed into aluminium oxide when exposed to electrolyte solution yielding OH- hydroxide ions [38]:

$$2Al + 6OH^- \rightarrow Al_2O_3 + 3H_2O + 6e^- \quad (1)$$

The resulting oxide layer thickness grows with anodizing time. It may amount to several dozen mm for protective and decorative layers or even exceed 100 mm for durable engineering

application aluminium layers. The final structure of layers depends on the alumina-solubility of the electrolyte solution [39].

In the initial stages of aluminium oxide film formation, a dense thin layer (0.01 – 0.1 μ*m*) of Al_2O_3 is formed (the so-called barrier or blocking layer), which subsequently changes into a porous layer as a result of barrier layer reformation at the oxide-electrolyte interface. The barrier layer is generated owing to Al^{3+} ions' migration in the electric field and their reaction with O^{2-} or OH^- ions producing anhydrous Al_2O_3. The final stage consists in the porous layer's expansion in thickness (up to 100 μ*m*) [36].

Oxide films obtained from poorly soluble solutions are characterized by a specific porous structure. Regularly distributed pores almost throughout the oxide layer and perpendicular to the surface have a diameter ranging from several to several tens of nanometres, depending on the conditions of anodic oxidation. The barrier layer is a thin non-porous layer situated from the inside. Due to the fact that the pores are densely distributed and small in diameter, the surface of such aluminium oxide is well-developed and is characterized by high adsorptivity, which is used in, e.g., introducing dyes.

For protective and decorative applications, it is the sulphuric acid bath which is the most commonly used. The layer thickness is adjusted according to norms depending on the operating conditions of the product [40].

The characteristic porous structure of oxide layers formed in the process of anodizing in sulphuric acid facilitates subsequent chemical dyeing operations. Organic dyes are frequently applied, most of which easily adsorb on the developed aluminium oxide surface. The process is usually performed in diluted dye solution at elevated temperature and in adjusted dye solution pH. The process, however, has one disadvantage, namely the poor light-fastness of the resulting surface colouring, particularly when exposed to direct sunlight. That is why only a small number of organic dyes are permitted for dyeing aluminium architectural elements [38, 41].

Chromate coatings are recognized as conversion coatings, i.e., coatings generated as a result of the chemical or electrochemical reaction of a metal surface layer with certain chemical compounds in which the produced salt is practically insoluble in the medium where the reaction is conducted [42, 43].

The previous study [44] provides results of the application of surface treatment operations on aluminium alloy sheets: yellow and black anodizing in sulphuric acid, self-colour anodizing and chromate conversion coating lead to certain changes in the surface free energy values. However, different variants of anodizing in sulphuric acid resulted in changes in percentage distribution of the dispersive and the polar component of the surface free energy. The results of the surface free energy and its component calculations indicate that the highest surface free energy value was obtained in the case of chromate coating (69.8mJ m^{-2}) and self-colour anodizing in sulphuric acid (68.5mJ m^{-2}). Simultaneously, the lowest value of the surface free energy was observed for black anodization.

A summary of the previous studies of selected surface treatment of various adherends are presented in Table 2.

No.	Type of surface treatment	Type of adherend
1	Degreasing	aluminium [12]
2	Mechanical treatment	aluminium [6, 30], steel [2, 32], titanium [4]
3	Chemical treatment	polymers [23], titanium [4]
4	Electrochemical treatment: anodizing, chromate coating	aluminium [24, 27, 39, 41-44]
5	Others: plasma treatment	aluminium [47]

Table 2. The examples of type of surface treatment and adherends

Numerous studies [2, 5, 7, 23-26] stress the importance of the effect of surface pretreatments for adhesive bonding and their effect on adhesive joint strength and the quality of adhesively bonded joints.

2.3. Surface preparation methods and adhesive joint strength

2.3.1. Adhesive joints: Types and dimensions

The research subject was a shear-loaded single lap adhesive joint of selected structural materials. Sample dimensions are presented in Figure 1: length of adherends l = 100±0.4 mm, width b = 20±0.3 mm, bond-line thickness: gk = 0.1±0.02 mm [46].

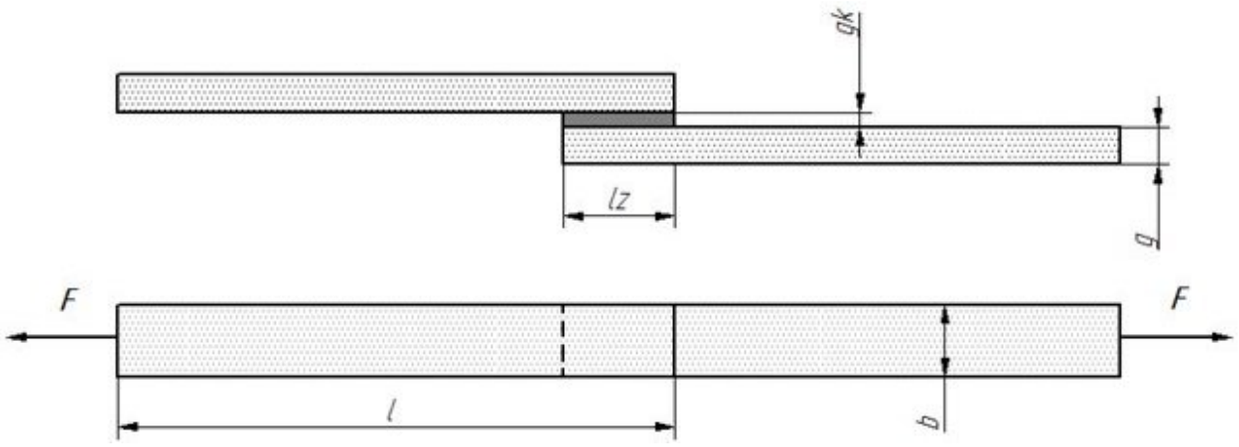

Figure 1. Single lap adhesive joint: b – adherends width, g – adherends thickness, g_k – bond-line thickness, l – adherends length, l_z – overlap length, P – force representing load type and direction

Thickness of adherends *g* and overlap length l_z, are presented in Table 3 [46].

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No.	Type of adherend	Adherend thickness g, mm	Overlap length l_z, mm
1	EN AW-2024PLT3 aluminium alloy sheet	0.64	24
2	EN AW-7075PLTO aluminium alloy sheet	0.80	27
3	CP1 titanium sheet	0.40	8
4	CP3 titanium sheet	0.80	16
5	X5CrNi181 stainless steel	1.00	14

Table 3. Dimensions of analysed adhesive joints

2.3.2. Adhesive joints forming

Adhesive joints were formed with a two-component epoxy adhesive, Loctite 3430 [47], suitable for the analysed adherends, with a short cure time at room temperature. The adhesive was prepared with a static mixer and applied on one of the adherends, in accordance with the manufacturer's recommendations. Even bond-line thickness was ensured in preliminary research by, inter alia, selecting a suitable amount of adhesive and pressure while curing.

Proper surface treatments were selected according to the given structural material:

1. degreasing (chemical clearing) with degreasing agents,

2. mechanical treatment with abrasive tools ,

3. anodizing,

4. chromate treatment,

5. etching,

6. a combination of selected methods listed above.

Degreasing in experimental tests was carried out with Loctite 7036 degreasing agent, mostly containing aliphatic hydrocarbons. Chemical cleaning with Loctite 7063 was a three-stage process consisting in spraying the surface of adherends, removing the agent with a cloth, and after the final application of the degreaser leaving the sample to dry. Chemical cleaning took place at an ambient temperature of 20±2 °C, and in relative humidity of 32-40 % [47].

Since chemical cleaning is rarely sufficient for providing good adhesion, as previously mentioned, the sample preparation included an abrasive mechanical treatment stage, performed with abrasive paper, which is the most convenient material for mechanical treatment of adherends. This approach is selected because of its high efficiency and accessibility combined with low cost, and its uncomplicated and versatile application in various conditions. Another important advantage of this method is that it requires little effort to ensure the machined surface shows marks in no direction.

Mechanical treatment in experimental tests was carried out with P320 abrasive paper.

The last stage of this surface treatment operation, having treated the surface with abrasive paper, was to remove the remaining contaminants from the surface with Loctite 7036 degreasing agent.

Sulphuric acid anodizing was another surface treatment used in tests. Adherends were immersed in 180-200 g/l solution of sulphuric acid at the temperature of 10÷15 °C for 35 minutes. Afterwards, the samples were dyed yellow in a 40÷55 g/l solution of $K_2Cr_2O_7$, pH 4.5÷6.2, at the temperature of 90÷95 °C for 35 minutes.

Another batch of samples was subjected to chromate treatment, consisting of a 30 second immersion in a 5.5 g/l solution of $Na_2Cr_2O_7$, a 4 g/l solution of Na_2SO_4; 4.5-5.5 g/l solution of H_3BO_3 and a 1.5 ml/l solution of HNO_3, pH 1.4-1.6.

Finally, the last surface treatment applied in tests for comparison was etching in a 40-60 g/l aqueous solution of sodium hydroxide. The adherends were immersed for 3-4 minutes at 45-55 °C, subsequently rinsed with warm running water and finally left to dry.

For the sake of control, a part of the samples remained untreated. This allowed the determination of the actual impact of surface treatments on the surface free energy values of adherends.

Joint forming conditions were as follows:

- cure temperature 20±2 °C,
- relative humidity 32-40 %,
- pressure when curing 0.02 MPa;
- seasoned for 48 h at ambient temperature of 20±2 °C.

2.3.3. Strength tests

Adhesive joint tensile-shear strength tests were performed in accordance with EN DIN 1465 standard on Zwick/Roell Z100 and Zwick/Roell Z150 testing machines. Testing speed was equal to 5 mm/min.

2.3.4. Test results

The results of shear strength tests on EN AW-2024PLT3 aluminium alloy sheet adhesive joints are presented in Figure 2. The results presented in figures are mean values of 8-2 measurements performed for each surface treatment variant.

Application of anodizing and chromate operations as an EN AW-2024 aluminium alloy sheet surface treatment method produced adhesive joints of maximum shear strength of 11.09 MPa and 12.39 MPa, respectively. Similarly good results in promoting joint strength were observed when the surface of EN AW-2024PLT3 aluminium alloy sheets was subjected to exclusively mechanical treatment; the results in that case amounted to 8.43 MPa and 8.66 MPa; therefore, the joint strength was six times higher than in the case where no surface treatment was applied. In the failure of EN AW-2024PLT3 aluminium alloy sheet adhesive joints, a characteristic and

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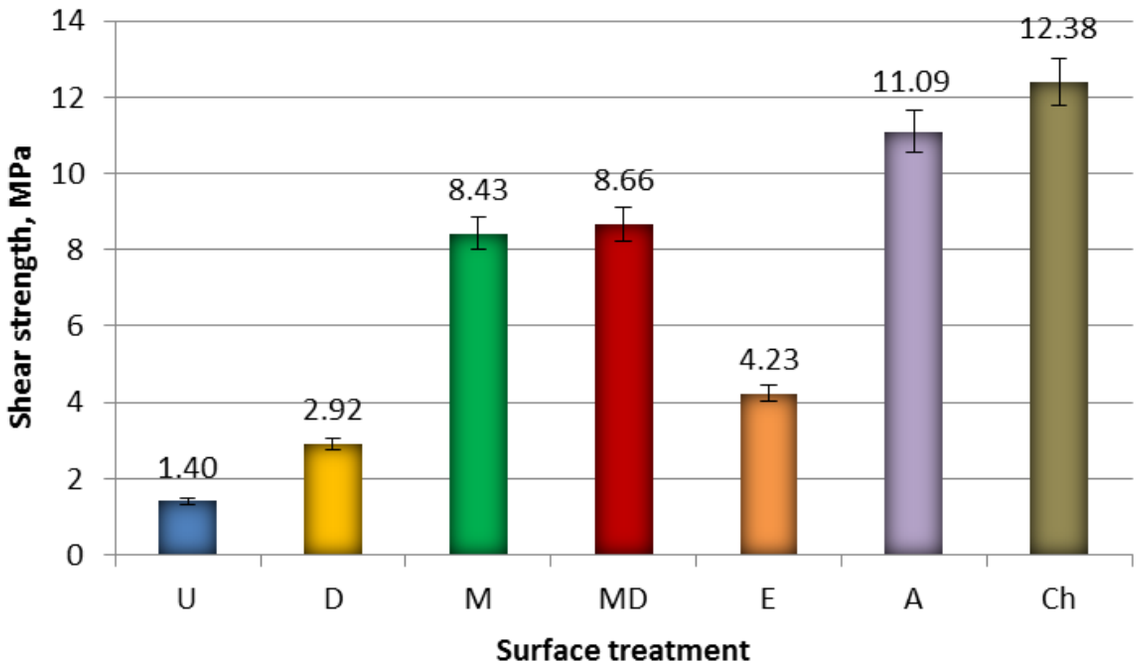

Figure 2. Shear strength tests of EN AW-2024PLT3 aluminium alloy sheet adhesive joints after different surface treatment methods: U - untreated, D - degreasing (chemical cleaning), M - mechanical treatment, MD - mechanical treatment and degreasing, E - etching A – anodizing, Ch - chromate treatment

repeatable shape of failed elements can be noticed. An observed plastic deformation of adherends results from shear and bending stresses.

Test results for homogenous EN AW-2024PLT3 aluminium alloy sheet adhesive joints' strength after certain surface treatment procedures are presented in Figure 3.

Adhesive joints were formed on 0.80 mm thick sheet samples. The highest sheet adhesive joint strength was observed after mechanical treatment (5.75 MPa), while the lowest was produced in the case of untreated adherends (4.30 MPa). In the latter case (variant M), the increase in joint strength was equal to 25 % as compared with variant U (untreated surface). The analysis of chemical treatment leads to the conclusion that the application of this particular operation generates conditions promoting joint strength. In addition, a positive impact of degreasing on joint strength can be observed in relation to the variant with no chemical treatment (approx. 9 % higher). It was observed that in each case when preparation of adherends' surface for adhesive joining was performed, higher joint strength is produced in comparison with variant U (untreated surface).

Adhesive joint strength tests were carried out on two types of titanium adherend: CP1 and CP3. CP1 titanium sheet adhesive joint strength after the analysed surface treatments is presented in Figure 4.

This shows that the highest strength was demonstrated by samples subjected to degreasing, and mechanical and degreasing. The shear strength of these joints was nearly four times higher than in the case of an untreated surface, whereas exclusively mechanical treatment only proved

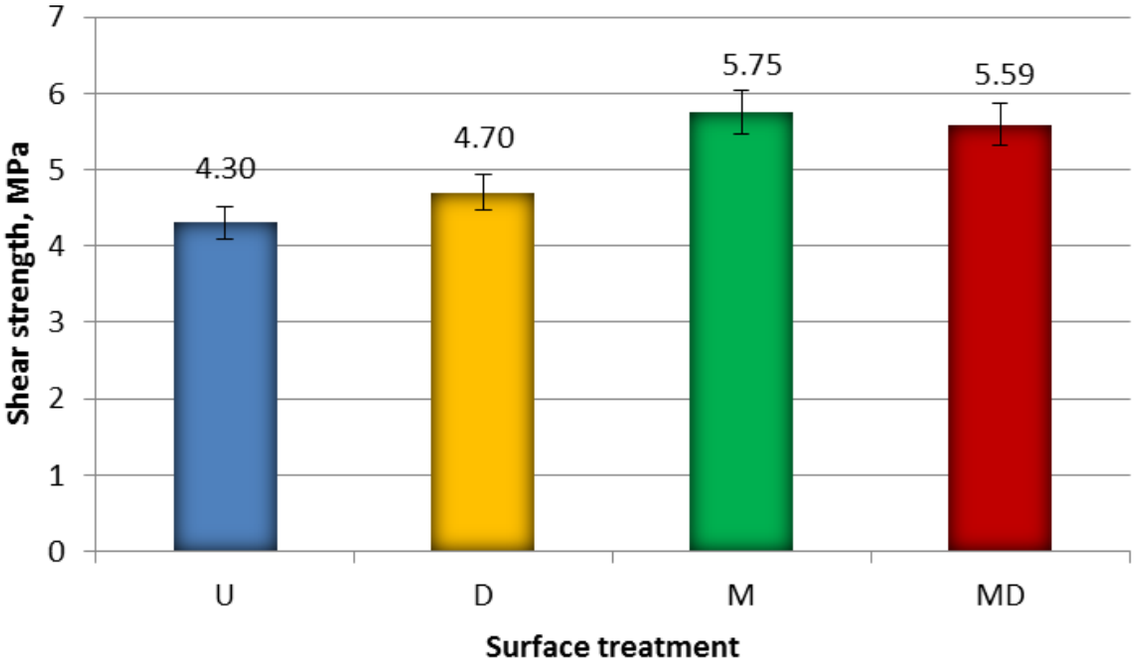

Figure 3. Shear strength tests of EN AW-7075PLT0 aluminium alloy sheet adhesive joints after different surface treatment methods: U - untreated, D - degreasing (chemical cleaning), M - mechanical treatment, MD - mechanical treatment and degreasing

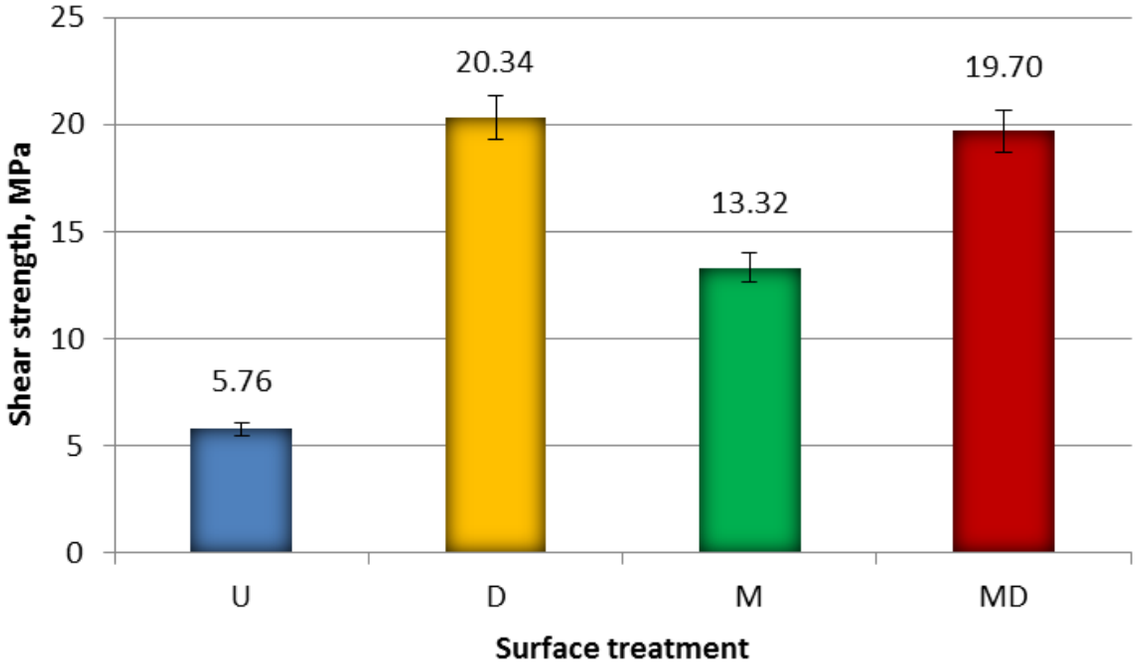

Figure 4. Shear strength tests of CP1 titanium sheet adhesive joints after different surface treatment methods: U - untreated, D - degreasing (chemical cleaning), M - mechanical treatment, MD - mechanical treatment and degreasing

slightly less effective. The shear strength developed here was notably lower by 7 MPa as compared to variant D (degreasing).

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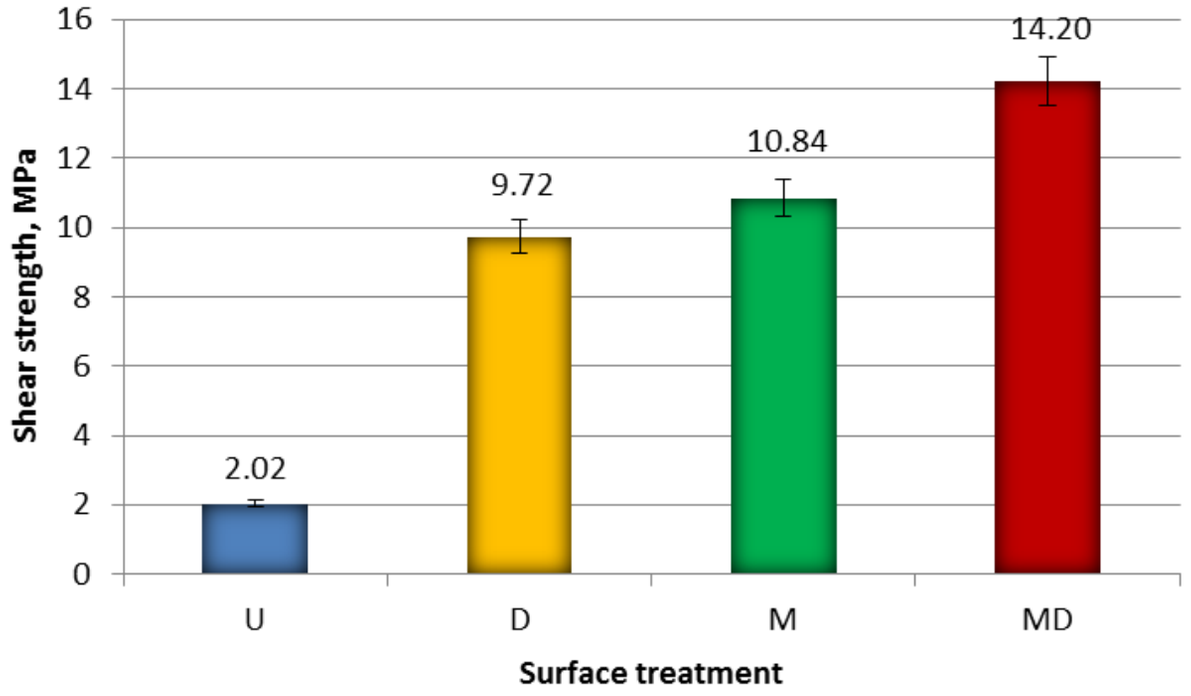

Figure 5. Shear strength tests of CP3 titanium sheet adhesive joints after different surface treatment methods: U - untreated, D - degreasing (degreasing), M - degreasing, MD - mechanical treatment and degreasing

Strength test results for CP3 titanium sheet adhesive joints after the analysed surface treatment are shown in Figure 5.

The application of mechanical and chemical treatment as surface treatment operations for CP3 titanium sheets was translated into the highest joint strength (14.20 MPa). The shear strength of such joints was seven times greater than in the case of an untreated surface. When treated mechanically, with no chemical cleaning, or in the case where only mechanical cleaning was applied, the resulting joint shear strength was lower. Homogeneous CP3 titanium sheet adhesive joint shear strength after degreasing constituted 68 % of strength value obtained following variant MD of surface preparation and 76 % of variant M (mechanical treatment).

Stainless steel adherend samples were treated in an identical manner to the case of aluminium and titanium sheet adherends. Joint strength test results after the analysed surface treatment are presented in Figure 6.

It was observed that the highest values of joint strength were obtained after mechanical and chemical treatment (14.84 MPa), but it was observed that there were large differences in the obtained test results. Standard deviation is significantly greater than for other types of surface treatment. The difference in the values of the strength after the application of degreasing and after machining and degreasing is about 10 %. Based on the results of the statistical analysis, it can be seen that the use of both degreasing and mechanical treatment makes it possible to obtain the same strength, with a higher reproducibility for the degreasing. Comparing the results of the bond strength after the surface treatment and without treatment by test materials

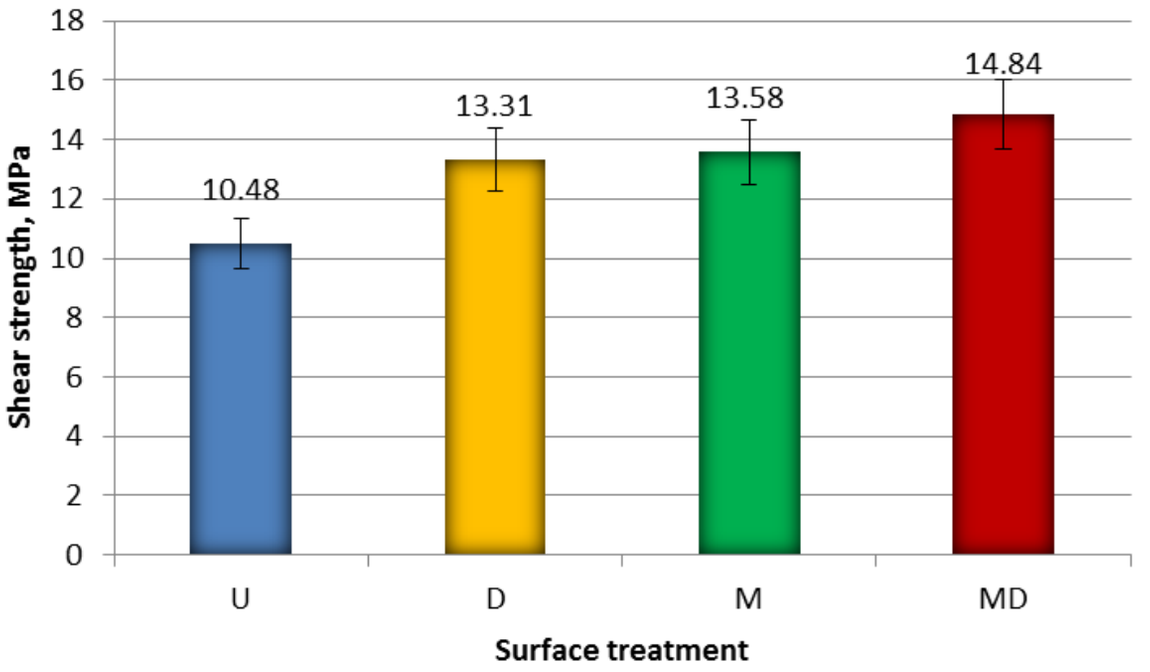

Figure 6. Shear strength tests of stainless steel adhesive joints after different surface treatment methods: U - untreated, D - degreasing (chemical cleaning), M - mechanical treatment, MD - mechanical treatment and degreasing

to prepare the surfaces for bonding, it can be noted that each of the analysed types of surface treatment allows for a greater strength to be obtained than the variant of untreated surface.

In numerous instances, when degreasing follows mechanical treatment, the resulting joint strength is higher as compared to joint strength of materials subjected exclusively to mechanical treatment. These results are evident in the case of CP1 and CP3 titanium sheet adhesive joints, while in EN AW-2024PLT3 and EN AW-7075PLTO aluminium alloy sheets the increase was insignificant. It ought to be mentioned, however, that chemical cleaning produced the highest scatter of joint strength value results as compared with other surface treatment operations. It could be indicative of higher non-uniformity of adhesive properties obtained in the case in question, which would in turn result in considerable differences in adhesive joint strength values obtained in tests. This observation is, furthermore, confirmed by the SFE value analysis carried out after degreasing of, e.g., CP1 (Figure 4) and CP3 (Figure 5) titanium sheets.

3. Adhesive properties, wettability and surface free energy

3.1. The characteristics of surface properties

Adhesive properties of the surface layer of structural materials determine the adequacy of the constituted surface layer for the processes where adhesion plays an essential role [48, 49].

Adhesive properties can be described with different physical quantities: the contact angle Θ and related wetting phenomenon, the work of adhesion W_a and the surface free energy. The

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contact angle Θ is an indicator of wettability – good wettability is marked by a small contact angle (Θ < 90°), whereas poor wettability co-occurs with a high contact angle (Θ > 90°) [50-52].

The small contact angle of water is presented in Figure 7 and the contact angle of diidomethane is presented in Figure 8. Figure 9 presents the high contact angle of water (Θ > 90°). Direct measurement of the contact angle of a liquid drop on the analysed surface is presented in Figures 7-9.

Many researchers argue that adhesive properties can be determined with surface free energy (SFE). This thermodynamic quantity describes the surface energetic state and is characteristic of particular solids or liquids. There are a number of SFE calculation methods, e.g., the Fowkes method, the Zisman method, the Owens-Wendt method and the van Oss-Chaudhury-Good method, the Neumann method and the method of Wu [49, 43-56].

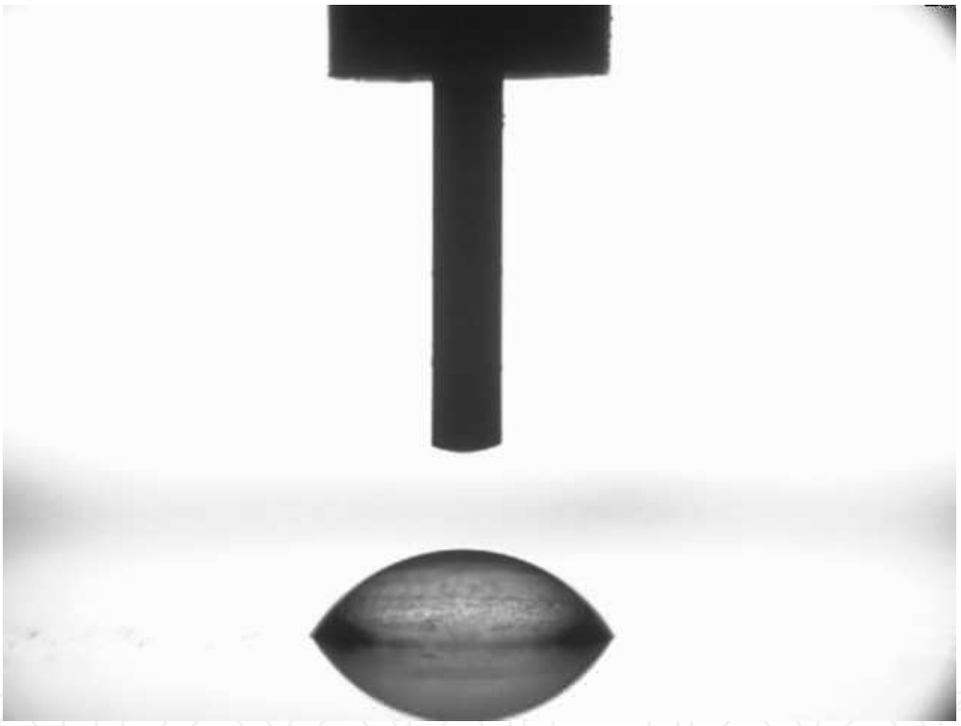

Figure 7. The small contact angle of water (Θ < 90°)

Numerous methods for direct measurement of the surface free energy are applied in liquids; however, in solids only indirect methods for determining the SFE can be applied.

3.2. Surface free energy after various surface treatments

3.2.1. Characteristics of tested materials and surface treatment

Tests were conducted on the following types of material:

- EN AW-2024PLT3 aluminium alloy sheets,

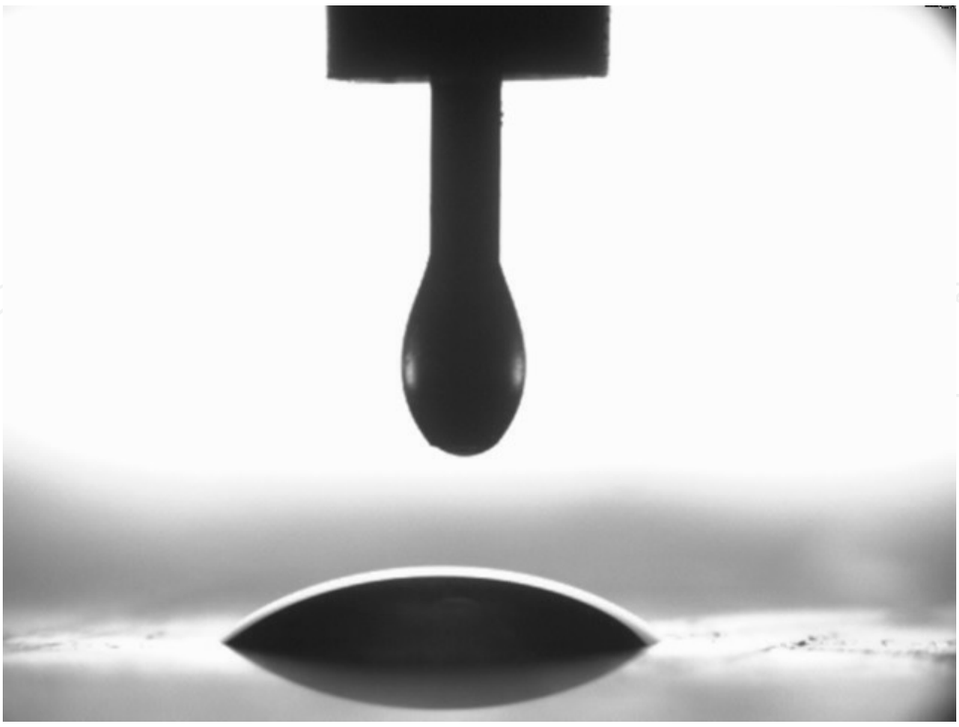

Figure 8. The small contact angle of diidomethane (Θ < 90°)

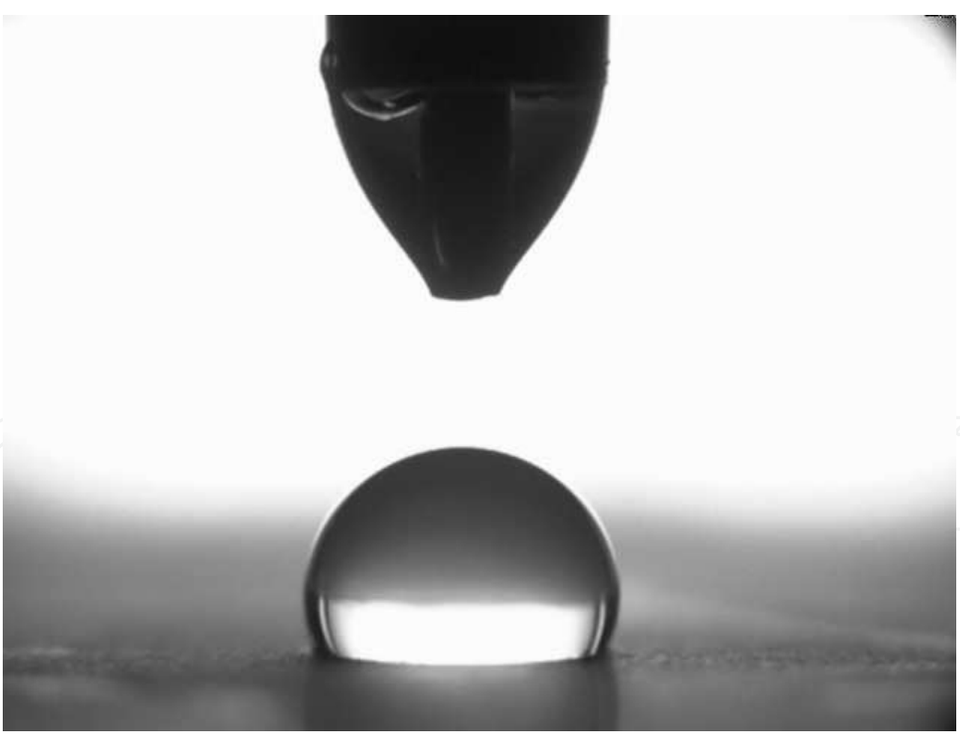

Figure 9. The high contact angle (Θ > 90°)

- EN AW-7075PLTO aluminium alloy sheets,

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- CP1 titanium sheets,
- CP3 titanium sheets,
- X5CrNi181 stainless steel.

Surface treatment operations were selected according to a given structural material [47]:

1. Degreasing with degreasing agents,
2. mechanical treatment with abrasive tools
3. anodizing,
4. chromate treatment,
5. etching,
6. a combination of selected methods listed above.

3.2.2. Method for determining free surface energy

Adhesive properties were determined by free surface energy. Free surface energy, in turn, was determined by the Owens-Wendt method. The method assumes that free surface energy (γ_S) is a sum of two components: polar (γ_S^p) and dispersive (γ_S^d), and that they all are characterized by the following dependence [5, 12, 22]:

$$\gamma_S = \gamma_S^d + \gamma_S^p \quad (2)$$

The polar component is defined as a sum of components generated by intermolecular forces, including polar, hydrogen, inductive, acidic and basic, excluding dispersive forces. Dispersive forces, on the other hand, are components of free surface energy. To determine polar and dispersive components of free surface energy, it is necessary to measure wetting angles of the surfaces of the materials being tested using two measuring liquids. Measuring liquids used to this end are liquids whose free surface energy and its polar and dispersive components are known. One of the liquids is apolar, while the other is bipolar. Distilled water was used as the bipolar liquid and diiodomethane was used as the apolar liquid. The components γ_S^d and γ_S^p of the tested materials can be determined using the relevant formulas given in the studies [12, 22].

The essential values of the applied measuring liquids' surface free energy γ_S and its components are listed in Table 4.

To calculate free surface energy, the wetting angle Θ of the surfaces of the tested materials was measured. The measurements were made using the method for direct measurement of the angle formed by a measuring liquid drop and the surface being examined. The measurements of the wetting angle were performed at a temperature of 26±2 °C and air humidity of 30±2 %. The volume of drops of the measuring liquids ranged from 0.8 to 1.5 μl. To every sample surface (there were 10 samples for each material), five drops of the measuring liquids were applied.

No.	Measuring liquid	γ_L [mJ/m²]	$\gamma_L{}^d$ [mJ/m²]	$\gamma_L{}^p$ [mJ/m²]
1	Distilled water	72.8	21.8	51.0
2	Diiodomethane	50.8	48.5	2, 3

Table 4. Values of free surface energy γ_L of the measuring liquids applied and its components [22]

After that, 5-10 measurements were made and the mean for each sample batch was calculated. The wetting angle was measured immediately following the application of a drop of the measuring liquid (after a few seconds). The measurements were made using a PGX goniometer manufactured by Fibro System (Sweden) and a PG programme for computer image analysis.

3.2.3. Results of surface free energy

The preparation of adherends for bonding was material-dependent, i.e., certain treatments, such as anodizing, chromate treatment or etching, were only applicable for 2024 aluminium alloy sheet adherends. The remaining samples were subjected to standard operations, mainly degreasing, mechanical treatment and the combination of the two. Figure 10 and Figure 11 present the values of the surface free energy γ_S and its components for the analysed aluminium alloys after particular surface treatments.

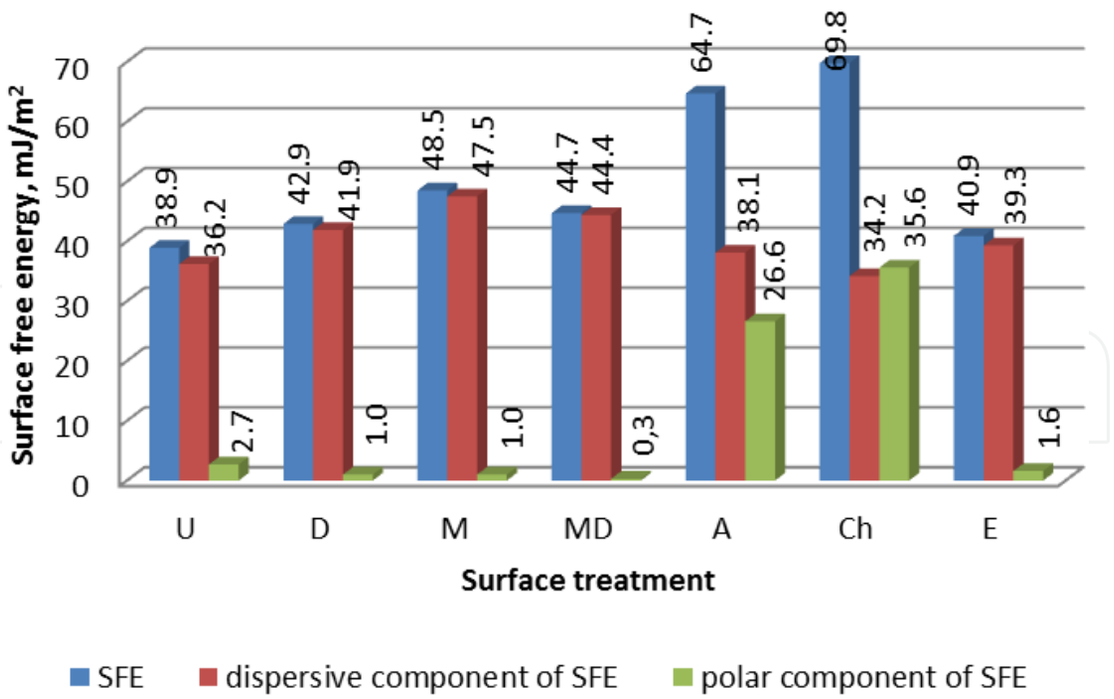

Figure 10. Surface free energy of EN AW-2024PLT3 aluminium alloy sheets: U – untreated, D – degreasing (chemical cleaning), M – mechanical treatment, MD – mechanical treatment and degreasing, A – anodizing, Ch – chromate treatment, E – etching

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Figure 12 and Figure 13 present the values of the surface free energy γ_{Sp} and its components for titanium sheets after particular surface treatments.

Figure 14 presents the values of the surface free energy γ_S and its components for stainless steel after particular surface treatments.

After analysis of the test results presented in Figures 10-14, it becomes apparent that there is a direct correlation between different surface treatments and the surface free energy of adherends. The final values of γ_S, however, depend on the type of material rather than the type of surface treatment. Furthermore, the values of polar γ_S^p and dispersive γ_S^d components of the surface free energy exhibit the tendency to vary not only within the same type of material but within the same type of surface treatment as well. In each of the analysed instances (excluding anodizing, Table 2.15) the γ_S^d component significantly dominates over the γ_S^p in total γ_S. It can be nevertheless noted that although anodizing produces a higher polar component of the surface free energy, γ_S component values are comparable.

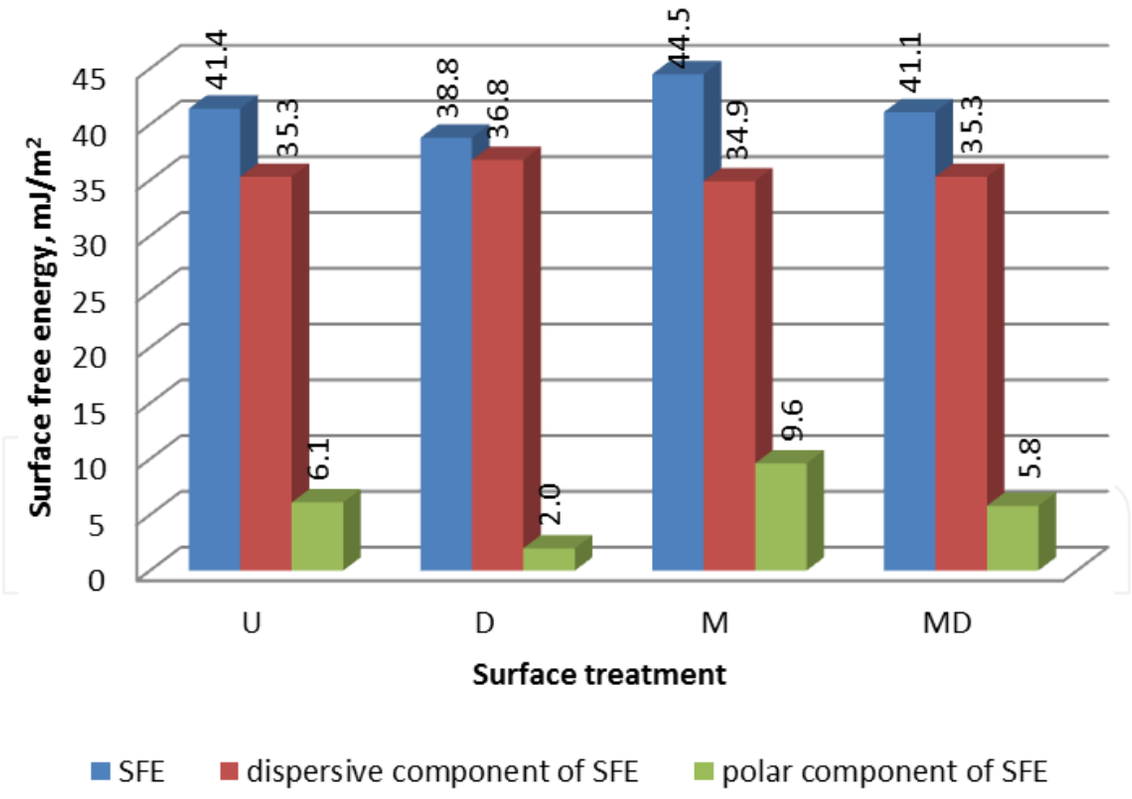

Figure 11. Surface free energy of EN AW-7075PLTO aluminium alloy sheets: U – untreated, D – degreasing (chemical cleaning), M – mechanical treatment, MD – mechanical treatment and degreasing

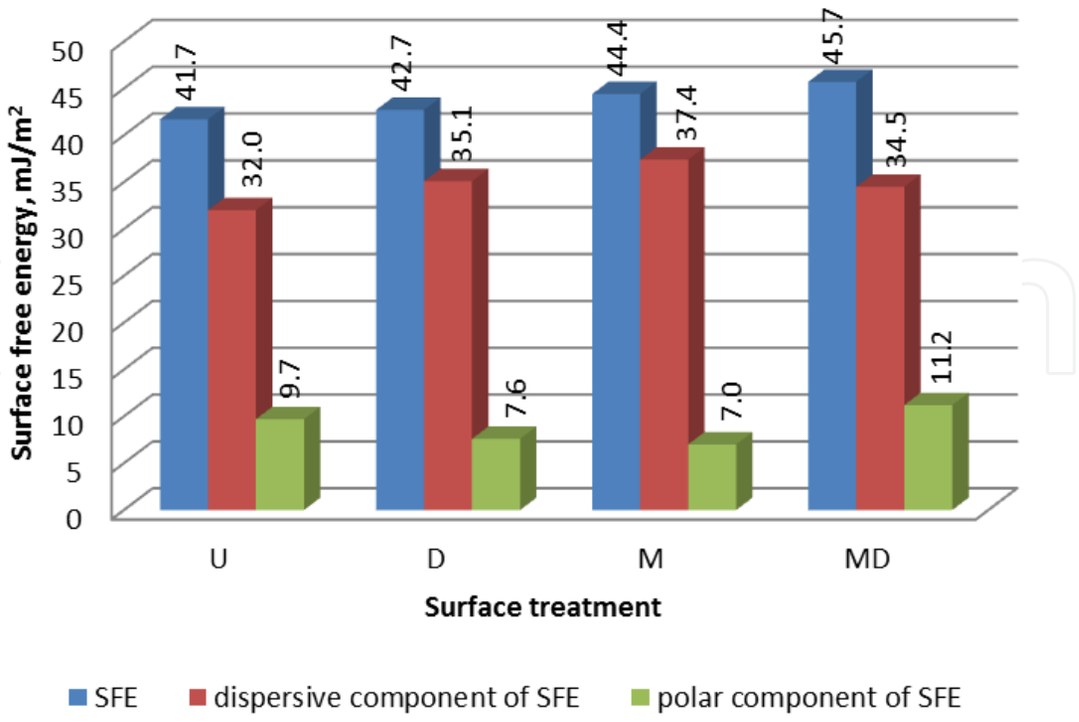

Figure 12. Surface free energy of CP1 titanium sheets: U – untreated, D – degreasing (chemical cleaning), M – mechanical treatment, MD – mechanical treatment and degreasing

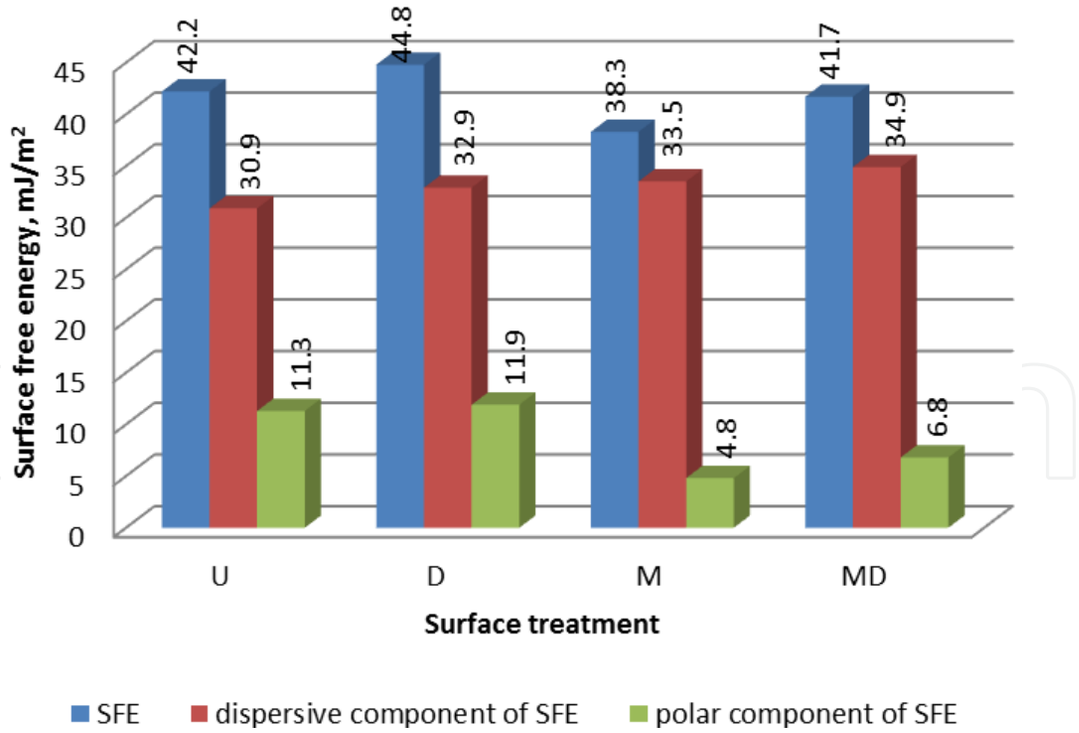

Figure 13. Surface free energy of CP3 titanium sheets: U – untreated, D – degreasing (chemical cleaning), M – mechanical treatment, MD – mechanical treatment and degreasing

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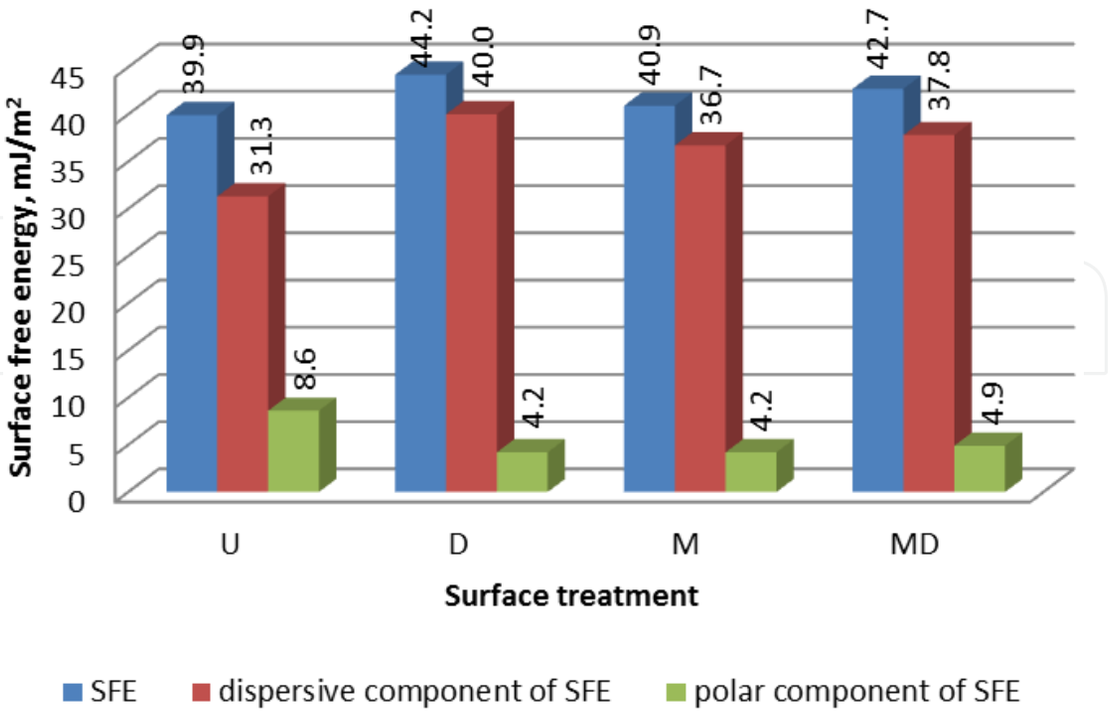

Figure 14. Surface free energy of stainless steel: U – untreated, D – degreasing (chemical cleaning), M – mechanical treatment, MD – mechanical treatment and degreasing

Bearing in mind the frequently negligible differences between the values of the surface free energy of different adherends after different surface treatments, the results were subjected to statistical analysis [46].

4. Geometric structure of adherends

4.1. The characteristics of geometric structure

The geometric structure of substrate surfaces is of considerable significance from the perspective of adhesive bonding. According to the mechanical theory of adhesion, penetration of micropores in adhered elements is an essential condition to be fulfilled in order for the mechanical interlocking to bear loads. The mechanical theory of adhesion recognizes different factors contributing to increasing adhesive joint strength [2, 3, 5, 21]. One of these dependencies is that with increased surface roughness of a given material, the number of irregularities which can be penetrated by the adhesive grows. This leads to the conclusion that the strength of adhesive joints formed on porous substrates is significantly higher than in the case of smooth-surface adherends, resulting from a considerably larger contact surface [3].

Excessive numbers of narrow micropores may hinder adhesive penetration, particularly in the case of high viscosity adhesive or that of high surface tension, where the adhesive may stop at the peaks of irregularities. In such a situation, micropores tend to trap air bubbles to form an additional weak boundary layer, acting to the detriment of adhesion. Therefore, there exists

a degree of surface roughness the exceeding of which produces disadvantageous conditions for the intermolecular bonds between adhesive and substrate.

The literature [2, 3, 23] presents dependencies used to describe the penetration of adhesive into micropores and irregularities of the surface in adhesive bonding. Depth of penetration is contingent on several factors, e.g., diameter of pores, viscosity, the surface free energy of adhesive or the wetting angle.

Different studies have analysed phenomena relevant to the relationship between surface roughness of adherends, their energetic state and wettability [32, 57, 58].

Mechanical treatment is one of the methods applied for the purpose of preparing the surface of adherends for bonding. Mechanical treatment consists in the removal of various surface contaminants, e.g., corrosion layers, and in addition, it enables surface development by constituting the geometric structure of adhered surface.

Of the numerous mechanical pretreatment methods, some prominent examples that could be mentioned are: sand- or grit-blasting, grinding or using coated abrasives [32, 58, 59]. Studies of the correlation between surface preparation and adhesive joint strength rarely offer detailed analysis of the impact of surface roughness parameters on adhesively bonded joint strength [59]. Moreover, certain researchers [2, 3] argue that surface roughness profile parameters alone fail to produce a satisfactory description of the degree of surface development.

4.2. The characteristic of geometric structure tests

The surface tests presented here indicate diversification of the geometric structure of adherends with respect to mechanical adhesion. Geometric structure often determines penetration of the adhesive into the surface irregularities and might promote mechanical adhesion, which is of great importance to adhesive bond strength.

Surface profiles, obtained from, inter alia, profilometer measurements, reveal that chemical cleaning fails to modify the surface geometry of analysed materials. On the other hand, mechanical, chemical and electrochemical treatments do generate considerable changes which consequently promote adhesion. It is for that reason that surface images and profiles were used in the characterization of the surface after chemical cleaning and mechanical treatment. In the case of EN AW-2024 aluminium alloy sheet, profiles and images were obtained following all applied surface treatments, i.e., anodizing, chromate treatment and etching, whereas for CP3 titanium sheets they were obtained after etching. The tables collate the results of measurements of surface roughness parameters, which represent mean values of 10-12 repetitions for each parameter.

The roughness of specimens was qualified by the method for measuring contact roughness, using an M2 profilometer manufactured by Mahr. The surface view was obtained using NanoFocus μscan AF2.

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4.3. Results of geometric structure – Aluminium alloy sheets

Two types of aluminium alloy sheets were tested in this study: EN AW-2024 (according to EN AW-2024-AlCu4Mg1 [60]) and EN AW-7075 (according to EN AW-7075-ALZn5.5MgCu [60]). The tests were performed on samples of different thicknesses and tempers [61-63]. Thickness and temper characteristics of analysed materials are presented in Table 5 and Table 6.

No.	Type of material	Sheet thickness g, mm	Temper
1	EN AW-2024 aluminium alloy sheet	0.64	T3
2	EN AW-2024PL aluminium alloy sheet	0.64	T3
3.	EN AW-2024PL aluminium alloy sheet	0.64	TO

Designations [350]:

O - thermally treated to produce stable tempers to develop mechanical properties, as after annealing,

T3 – heat treated, subjected to cold working and natural ageing until reaching a stable condition,

PL – plated.

Table 5. Characteristics of EN AW-2024 aluminium alloy sheets

No.	Type of material	Sheet thickness g, mm	Temper
1	EN AW-7075PL aluminium alloy sheet	0.80	TO

Designations [350]:

O - thermally treated to produce stable tempers to develop mechanical properties, as after annealing,

PL – plated.

Table 6. Characteristics of EN AW-7075 aluminium alloy sheets

Table 7 and Table 8 present characteristic surface roughness profile parameters (average from 10-12 values) of the analysed aluminium alloys subjected only to degreasing (chemical cleaning).

No.	Type of aluminium alloy	Mean values of surface roughness parameters, μm						
		R_a	R_z	$R_{z\,max}$	S_m	R_p	R_{pk}	R_{vk}
1	EN AW-2024T3	0.28	2.10	2.50	70	0.90	0.34	0.37
2	EN AW-2024PLT3	0.20	1.61	2.80	208	0.77	0.28	0.44
3	EN AW-2024PLTO	0.60	4.10	5.30	70	2.56	1.04	0.65

Table 7. Surface roughness parameters of EN AW-2024 aluminium alloy sheets

No.	Type of aluminium alloy	Mean values of surface roughness parameters, μm						
		R_a	R_z	$R_{z\,max}$	S_m	R_p	R_{pk}	R_{vk}
1	EN AW-7075PLTO	0.15	1.12	2.06	203	0.54	0.22	0.41

Table 8. Surface roughness parameters of EN AW-7075PLTO aluminium alloy sheets

It was noted that it could be as a result of rolling that particular surface roughness values are obtained. In EN AW-2024 aluminium alloy sheets' temper O there was a marked increase in surface roughness as compared with EN AW-7075 aluminium alloy sheets' temper O.

The surface roughness parameter results of the analysed aluminium alloy sheets (after degreasing) are furthermore reflected by surface images and profiles from a 3D profiler. Representative sheet specimens of different tempers are presented in Figures 15-18 [44].

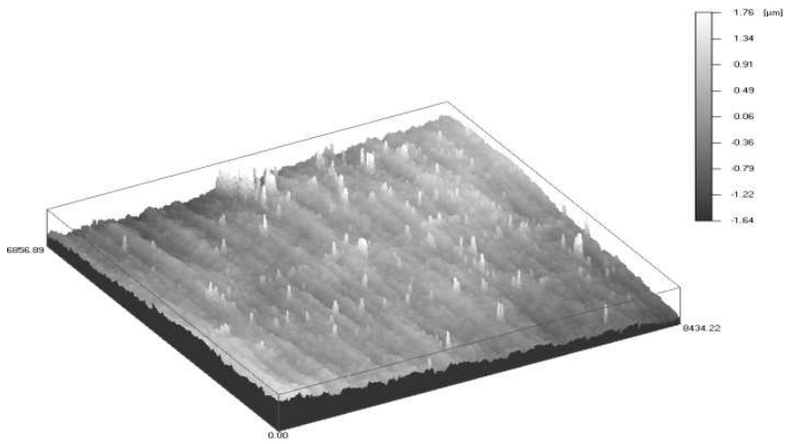

Figure 15. Surface of EN AW-2024PLT3 aluminium alloy sheet; degreasing, 3D profiler

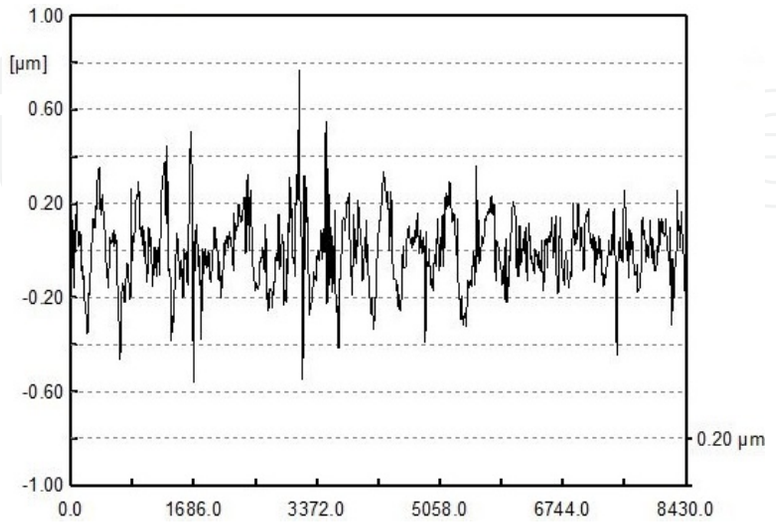

Figure 16. Representative profile of EN AW-2024PLT3 aluminium alloy sheets; degreasing

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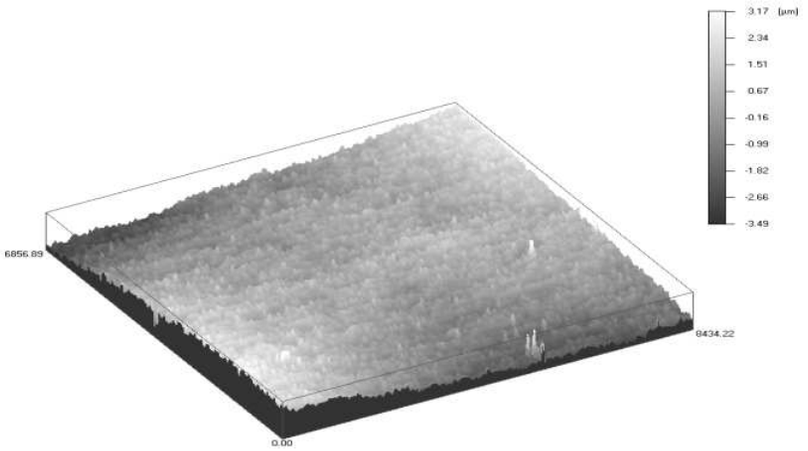

Figure 17. Surface of EN AW-7075PLTO aluminium alloy sheets; degreasing, 3D profiler

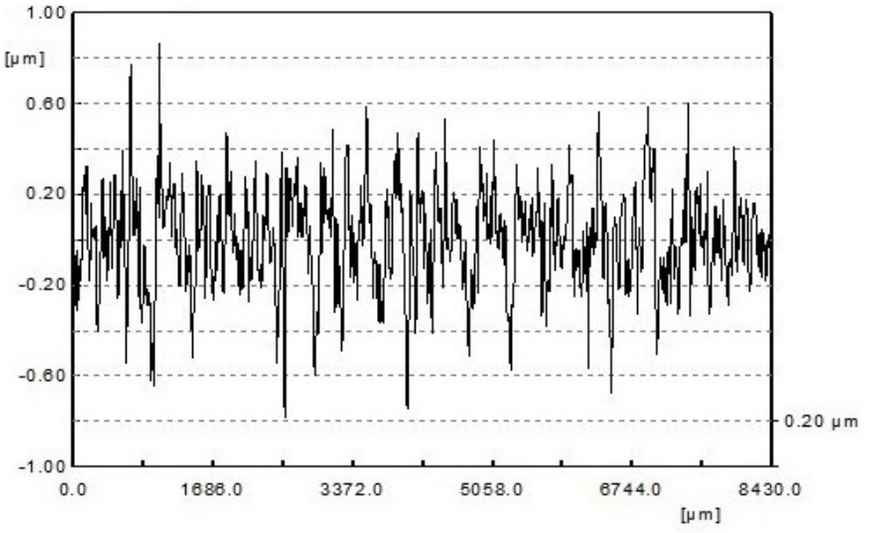

Figure 18. Representative profile of EN AW-7075PLTO aluminium alloy sheets; degreasing

In comparing images and profiles of the analysed surfaces, it emerges that it was probably the differences in the rolling process of the analysed sheets that produced differences in the geometric structure of the surface.

Surface roughness measurements of EN AW-2024PLT3 and EN AW-7075PLTO aluminium alloy sheets after different surface treatments are presented in Table 9 and Table 10.

As opposed to chemical cleaning, mechanical treatment did modify the geometric structure of aluminium alloy sheet surface. Surface roughness parameters of EN AW-7075PLTO following mechanical treatment and degreasing are decreased in comparison with samples subject to mechanical treatment only, which could be a consequence of removing mechanical treatment residue from the surface. Model images and roughness parameters (average from 10-12 values) after this surface treatment method are presented in Figures 19-22.

No.	Surface treatment	Mean values of surface roughness parameters, μm						
		R_a	R_z	$R_{z\,max}$	S_m	R_p	R_{pk}	R_{vk}
1	Chemical cleaning	0.20	1.36	2.59	270	0.65	0.28	0.64
2	Mechanical treatment	1.31	9.83	14.24	137	4.87	2.17	2.52
3	Mechanical treatment and chemical cleaning	1.40	9.58	12.46	125	4.44	2.00	2.73
4	Anodizing	0.41	3.78	5.79	122	1.49	0.52	1.02
5	Chromate treatment	0.37	3.48	5.70	108	1.52	0.73	0.77
6	Etching	0.33	2.72	4.63	145	1.04	0.48	0.87

Table 9. Surface roughness parameters of EN AW-2024PLT3 after different surface treatments

No.	Surface treatment	Mean values of surface roughness parameters, μm						
		R_a	R_z	$R_{z\,max}$	S_m	R_p	R_{pk}	R_{vk}
1	Chemical cleaning	0.31	2.31	4.64	290	1.13	0.64	0.72
2	Mechanical treatment	1.58	12.07	16.96	142	6.17	2.68	3.14
3	Mechanical treatment and chemical cleaning	1.28	9.16	12.12	113	4.41	1.79	2.53

Table 10. Surface roughness parameters of EN AW-7075PLTO after different surface treatments

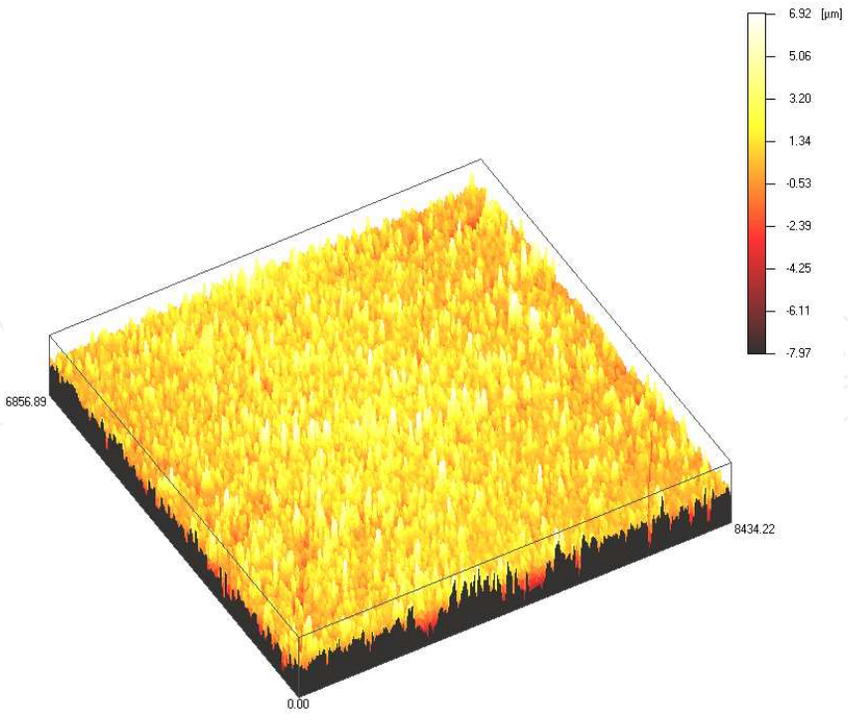

Figure 19. Surface of EN AW-2024PLT3 aluminium alloy sheets; mechanical treatment, 3D profiler

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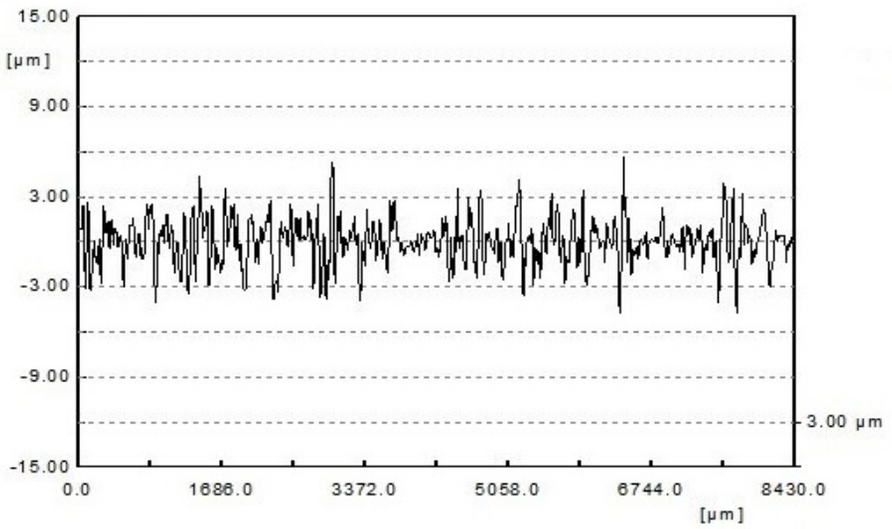

Figure 20. Representative profile of EN AW-2024PLT3 aluminium alloy sheets; mechanical treatment

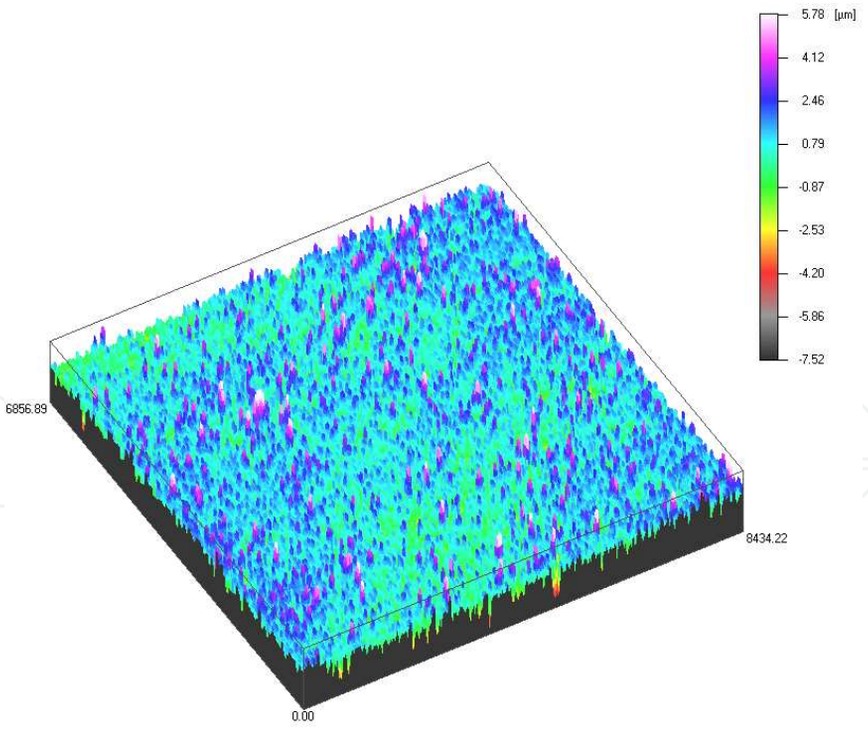

Figure 21. Surface of EN AW-7075PLTO aluminium alloy sheets; mechanical treatment, 3D profiler

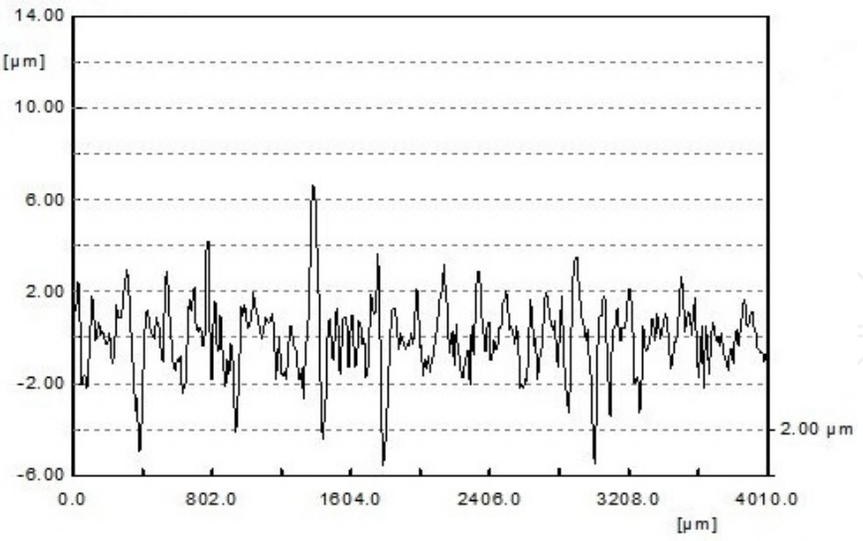

Figure 22. Representative profile of EN AW-7075PLTO aluminium alloy sheets; mechanical treatment

The surface of EN AW-2024PLT3 and EN AW-7075PLTO aluminium alloys after mechanical treatment manifests a considerably higher number of micro-irregularities in the uniform distribution on the surface (Figures 19 and 21). Simultaneously, an increase in surface roughness resulting from mechanical treatment (4-6 times) (Table 9 and Table 10), compared with most of the other surface treatments, should be noted.

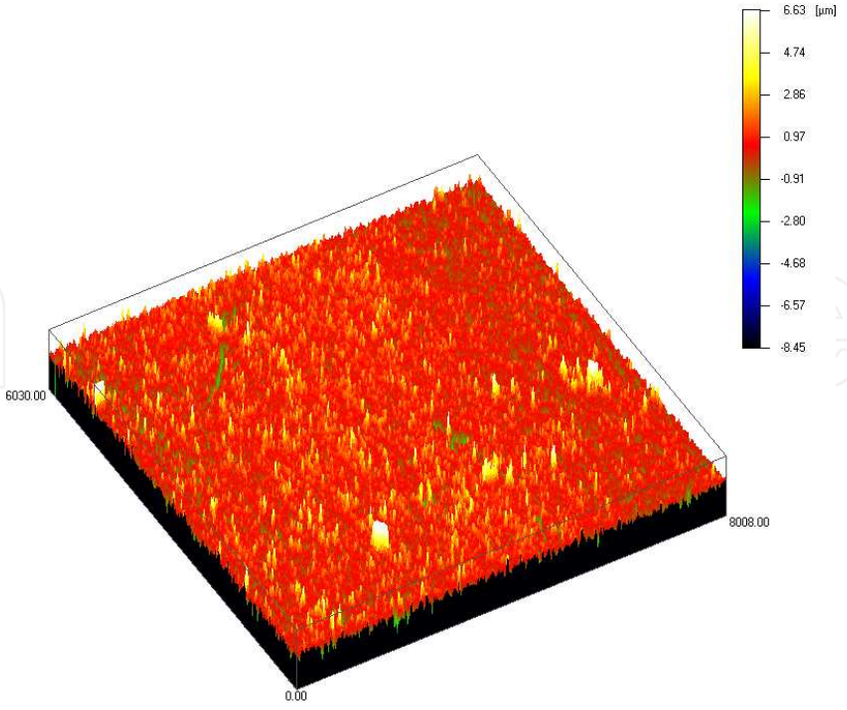

Figure 23. Surface of EN AW-7075PLTO aluminium alloy sheets; anodizing, 3D profiler

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The surface roughness parameter results for the analysed aluminium alloy sheets EN AW-2024PLT3 after anodizing, chromate treatment and etching are reflected by surface images and profiles from a 3D profiler. Representative sheet specimens are presented in Figures 23-28.

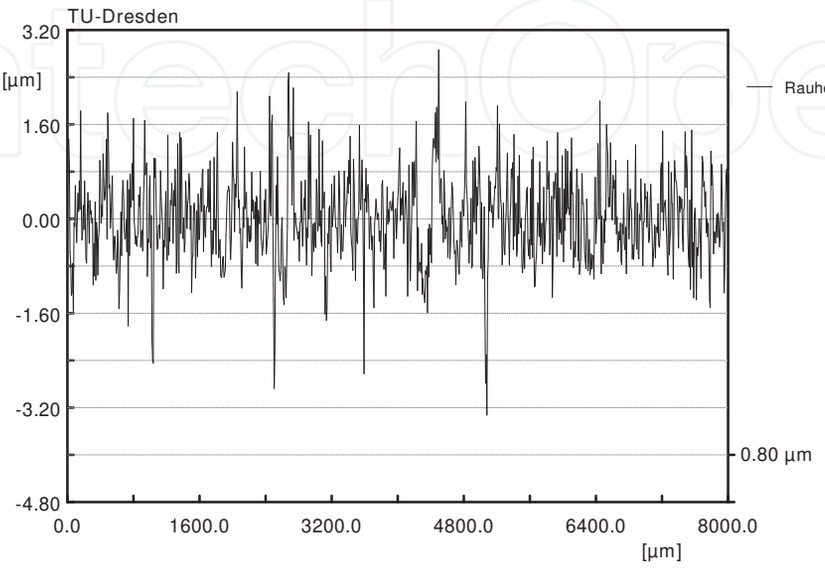

Figure 24. Representative profile of EN AW-2024PLT3 aluminium alloy sheets; anodizing

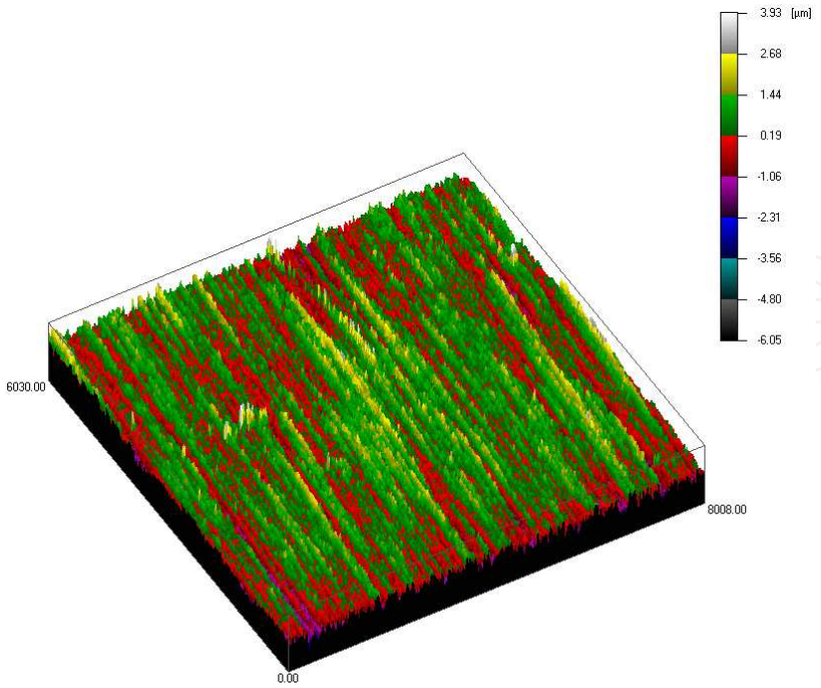

Figure 25. Surface of EN AW-2024PLT3 aluminium alloy sheets; chromate treatment, 3D profiler

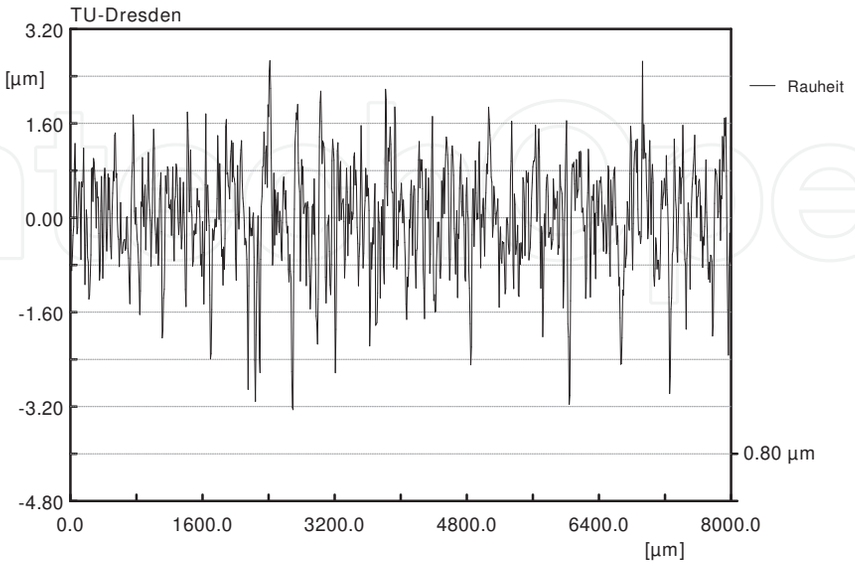

Figure 26. Representative profile of EN AW-2024PLT3 aluminium alloy sheets; chromate treatment [44]

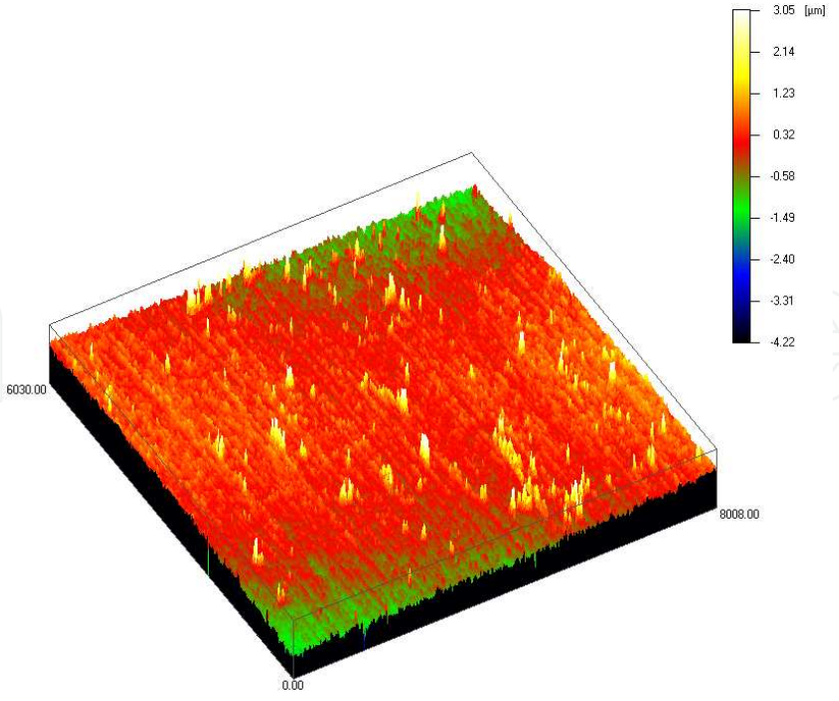

Figure 27. Surface of EN AW-2024PLT3 aluminium alloy sheets; etching, 3D profiler

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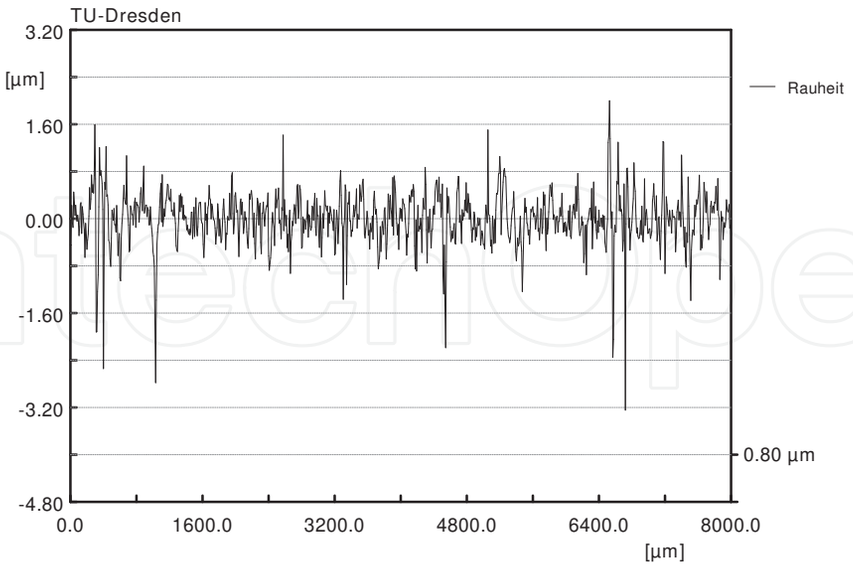

Figure 28. Representative profile of EN AW-2024PLT3 aluminium alloy sheets; etching

The results of surface roughness parameter measurement carried out on test samples subjected to anodizing indicate that anodizing has an impact on the height of surface irregularities.

Comparing analysed samples subjected to surface treatment technologies, it was chromate conversion coating which proved to have the greatest impact on both the height and the structure of surface irregularities, as shown in Figure 25 and Figure 26. In the case of this method, the decrease in the values of irregularities' height parameters ranged between -6.2 % for the *Rk* and -42.6 % for the *Rvk*, while there was practically no difference in the values of the two parameters *Rp* and *Rpk*. As a result, noticeable smoothing of the test sample surface was achieved. Reduced mean spacing of profile peaks' *Sm* by 18 % and the local *S* by 12.5 % together represent an increased number of irregularities, as confirmed by the representation of the roughness profile in Figure 26 and of the structure in Figure 25 [44].

5. Conclusions and summary

Results obtained from adhesive joint strength testing of the materials show that surface treatment plays an important role in increasing the strength of analysed joints. Moreover, the application of the same surface treatment in the context of different structural materials produces different strength values.

Tests indicate that in numerous instances it is mechanical treatment only or mechanical treatment followed by degreasing which translate into the highest joint strength. Statistical analysis of adhesive joint strength test results demonstrated that in certain cases no significant differences between different surface treatments effects can be detected. Moreover, degreasing

conducted after mechanical treatment is not reflected in a significant increase in joint strength values.

In certain joints, e.g., CP1 titanium sheets, degreasing is sufficient to provide relatively high joint strength. Furthermore, in certain applications, the differences between the effects of degreasing and mechanical treatment with degreasing showed no statistical significance. It was also noted that mechanical treatment can produce joints of substantial strength (e.g., EN AW-7075PLTO aluminium sheet adhesive joints).

The applied surface treatment methods, such as mechanical, chemical and electrochemical methods, modify geometry and roughness parameters. Chemical cleaning, however, has little impact on surface geometry, in the case of both untreated and mechanically treated surfaces. Nevertheless, in certain applications, change in surface parameters can be observed.

The surface treatment method that introduces extensive changes in the analysed materials' surface geometry is mechanical treatment. In some cases it has, moreover, played a role in increasing surface roughness parameters, e.g., in aluminium alloys and titanium sheets.

The application of various surface treatments in different structural materials allows for modification of their adhesive properties, determined by the surface free energy. Statistical analysis proved that in the majority of cases the surface free energy values responded considerably to the surface treatment operations. Although in some of the analysed variants the differences in γ_S values were negligible, they were nevertheless statistically relevant (level of significance of 0.05).

It was noted that different surface treatments contribute not only to the surface free energy changes but to the SFE components' share in the total value. In the majority of variants of EN AW-2024PLT3 aluminium alloy sheet surface treatment, the dispersive component amounted to the 93-99 % of the total surface free energy. Electrochemical treatment (anodizing and chromate treatment) produced very different results. The polar component γ_{S^p} after chromate treatment was greater than the dispersive component γ_{S^d}, and after anodizing the polar component amounted to 41 % of total SFE. It appears, then, that chemical treatment methods manifest capabilities to increase the surface free energy and to balance the dispersive-to-polar component ratio. This ratio is typical of particular technologies of surface treatment; for instance in CP1 and CP3 titanium sheets, the dispersive component of the SFE ranges between 73 % and 87 % of the total surface free energy, whereas for stainless steel it is 78 %-90 %.

With regard to the surface free energy values in EN AW-2024PLT3 aluminium alloy sheets, it is electrochemical treatment (chromate treatment and anodizing) which is the most advantageous. In the case of other treatments, the second-best mechanical treatment is additionally beneficial in EN AW-7075PLTO aluminium alloy sheets and CP1 titanium sheets.

Development of surface geometry and roughness parameters of the analysed adherends does not always correlate with the increase in the values of the surface free energy of these materials. It is a non-linear dependence, in which the increase of surface roughness parameters is on several occasions not reflected in an identical increase in the surface free energy.

The assumption, then, should be that in order for the determination of a particular surface for adhesive processes to be comprehensive, it should account for the adherend's surface geometry as well as its adhesion properties. The geometry of the surface can influence the mechanical adhesion, and the surface free energy is connected with both mechanical adhesion and the other constituent of adhesion: proper adhesion.

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